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# On the effect of misalignment on Bragg angles obtained from zero-layer Weissenberg X-ray photographs

V. K. WADHAWAN

*Nuclear Physics Division, Bhabha Atomic Research Centre, Trombay, Bombay*-85 (*AS*)

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An expression has been derived for the error introduced in Bragg angles obtained from zero-layer Weissenberg X-ray photographs when the crystal used is not perfectly aligned. The error increases monotonically with $\theta$ for $\theta > 45°$. This makes good alignment of crystals all the more important, as it is the high-angle reflections which are preferred for accurate determination of lattice parameters.

## INTRODUCTION

Bragg angles of reflections recorded in a zero-layer Weissenberg photograph can be obtained by measuring the heights of the corresponding spots from the base line In fact, for a camera of 57 3 mm diameter, a measurement of these ordinates in millimeters gives directly the values of Bragg angle $\theta$, in degrees. Such measurements are needed, for example, for determining the lattice parameters of crystals by a least-squares fitting procedure through the application of the Bragg law But if such a procedure is to yield accurate results, it is essential to take into account the effect of various systematic errors like those due to film shrinkage, beam divergence, eccentricity of specimen, absorption, misalignment, etc., by introducing correction terms in the expression for the calculated value of $\theta$. Expressions for such correction terms are available for most of the major sources of systematic error (Buerger 1942, Nelson & Riley 1945, Henry *et al* 1953, Nuffield 1966). In this paper an expression is derived for the effect of any residual amount of crystal misalignment on the values of Bragg angles obtained by measuring the ordinates of the reflection spots in a zero-layer Weissenberg photograph.

## CALCULATION OF ERROR CAUSED BY MISALIGNMENT

In figure 1(a), the circle with radius OR (equal to 1 reciprocal lattice unit) represents a central section of the sphere of reflection by a plane perpendicular to the axis of rotation. R is the origin of the reciprocal lattice (r.l.) and P a r.l. point in the zero-layer normal to the axis of rotation. If there is no misalignment present, the r.l. vector $RP$ would be at right angles to the axis of rotation and a reflection would flash out in the direction of OQ when, on rotating the crystal, the r.l. point P intersects the sphere of reflection at Q. The angle ROQ is equal to $2\theta$, and if S is the point where the reflected beam strikes the film placed coaxially with the axis of rotation through O, the distance BS measured along the arc would be proportional to $2\theta$, the proportionality being through an instrumental constant.

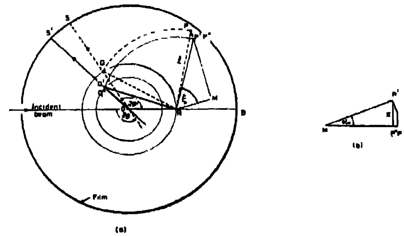


Figure 1. Geometry of the Bragg reflection process and the Weissenberg set-up.

Now suppose there is a certain amount of misalignment present in a general direction. The zero-layer will no longer be normal to the axis of rotation. The misalignment can be represented by an axial vector in the equatorial plane. Let the direction of this vector be along RM, and its magnitude $\alpha_0$. Also, let $\xi$ be the angle that the r.l. vector $RP$ makes with this vector. Unless $\xi = 0$, the r.l. point P will not now lie in the equatorial plane. Let P′ be its new position, at a perpendicular distance, say, $x$ from the equatorial plane To calculate $x$, we drop a perpendicular, PM, from P on RM. Also, let P″ be the point of projection of P′ on the equatorial plane, so that P′P″ $= x$ (figure 1(b)). P″ will lie on PM. If RP $= l$, then PM $=$ P′M $= l \sin \xi$. From figure 1(b), $x$ is then given by

$$x = l \sin \xi \sin \alpha_0. \tag{1}$$

From figure 1(b) again, we have P″M $= l \sin \xi \cos \alpha_0$. Going back to figure 1(a), since RM $= l \cos \xi$, if we denote RP″ by $l'$, we get

$$\begin{aligned} l' &= (\mathrm{RM}^2+\mathrm{P''M}^2)^{1/2} \\ &= (l^2 \cos^2\xi + l^2 \sin^2 \xi \cos^2\alpha_0)^{1/2} \\ &= l(1-\sin^2 \xi \sin^2 \alpha_0)^{1/2}. \end{aligned} \tag{2}$$

We now consider a section of the sphere of reflection by a plane parallel to the equatorial plane and at a distance $x$ from it, so that the point $P'$ lies on this plane. This section is a circle with a radius, $r'$, given by

$$r' = (1-x^2)^{1/2}$$

which, on using equation (1), becomes

$$r' = (1-l^2 \sin^2 \xi \sin^2 \alpha_0)^{1/2}. \quad \ldots \tag{3}$$

When the crystal is rotated, a Bragg maximum will now occur in the direction of OQ$'$, intersecting the film at S$'$. Since the observed value of Bragg angle, say $\theta'$, is taken as proportional to the distance BS$'$ measured along the arc, *i.e.*, as proportional to the ordinate of the spot on the flattened film, any shift in spot position in a direction parallel to the rotation axis is of no consequence Therefore, we can obtain the value of $\theta'$ by projecting the triangle ROQ$'$ on the equatorial plane. If Q$''$ is the point of projection of Q$'$, we have OQ$''$ $= r'$, RQ$''$ $= l'$ and OR $= 1$ for the triangle ROQ$''$. We can therefore write

$$\cos 2\theta' = \cos \widehat{ROQ''} = \frac{r'^2+1-l'^2}{2r'} .$$

On using equations (2) and (3) this becomes

$$\cos 2\theta' = \frac{2-l^2}{2(1-l^2 \sin^2 \xi \sin^2 \alpha_0)^{\frac{1}{2}}} \qquad \ldots \quad (4)$$

Now, $l$ is related to the true Bragg angle, $\theta$, through the Bragg condition as follow

$$l = \frac{n\lambda}{d} = 2 \sin \theta. \qquad \ldots \quad (5)$$

Substituting this in equation (4), we get

$$\cos 2\theta' = \frac{\cos 2\theta}{(1-4 \sin^2 \xi \sin^2 \alpha_0 \sin^2 \theta)^{\frac{1}{2}}} \qquad \ldots \quad (6)$$

Since the amount of misalignment generally present is very small, an expression for the error $(\theta'-\theta)$ can be obtained to a good approximation by making the following assumptions :

$$\left.\begin{aligned} \sin^2 \alpha_0 &\ll 1/4 \\ \sin^2 \alpha_0 &\sim \alpha_0^2 \\ \sin(\theta-\theta') &\sim (\theta-\theta') \\ \sin(\theta+\theta') &\sim \sin 2\theta \end{aligned}\right\} \qquad (7)$$

Equation (6) can be recast as

$$\cos 2\theta - \cos 2\theta' = \cos 2\theta[1-(1-4 \sin^2 \xi \sin^2 \alpha_0 \sin^2 \theta)^{-\frac{1}{2}}],$$

or,

$$-2 \sin(\theta+\theta') \sin (\theta-\theta') = \cos 2\theta[1-(1-4 \sin^2 \xi \sin^2 \alpha_0 \sin^2 \theta)^{-\frac{1}{2}}].$$

Making use of assumptions expressed in (7), we get

$$\theta'-\theta \approx \frac{\cos 2\theta}{4 \sin \theta \cos \theta}[1-(1+2\alpha_0^2 \sin^2 \xi \sin^2 \theta)],$$

which simplifies to

$$\theta'-\theta = -\frac{\alpha_0^2}{2} \sin^2 \xi \tan \theta \cos 2\theta. \qquad \ldots \quad (8)$$

The angle $\xi$ can be expressed as the sum of a fixed part and a variable part :

$$\xi = \delta_0 + \phi \qquad \dots \quad (9)$$

Here $\delta_0$ is the fixed angle between the misalignment vector and some reference direction; the referece direction can be, for example, one of the r.l. axes. And $\phi$ is the angle between the reference direction and the relevant r.l. vector, and varies from reflection to reflection. $\phi$ can expressed in terms of the r l. parameters and the Miller indices. The expression for the correction term for $\theta$ can finally be written as

$$\theta' - \theta = \left[ -\frac{\alpha_0^2}{2} \sin^2(\delta_0 + \phi) \right] \tan\theta \cos 2\theta \qquad (10)$$

In this expression, $\alpha_0$ and $\delta_0$ are the constants which specify the misalignment vector completely, and these can be treated as two of the independent parameters in a least-squares fitting procedure employed for refining the lattice parameters (Wadhawan 1972).

## Discussion

As expected, equation (8), as also the exact equation (6), show that the error $(\theta' - \theta)$ caused by a given misalignment $\alpha_0$ is the maximum for $\xi = 90°$. Again, these equations show that the error depends only on the magnitude of $\alpha_0$ and is independent of its sign. This again is only to be expected, because a reversal of the sign of $\alpha_0$ will simply mean a reversal in the *horizontal* shift of spot positions on the flattened film, the vertical shift, or the effect on ordinates, remaining unchanged.

Figure 2 shows the variation of $(\theta' - \theta)$ with $\theta$ for various degrees of misalignment. The curves have been obtained from equation (6), taking $\xi = 90°$. For

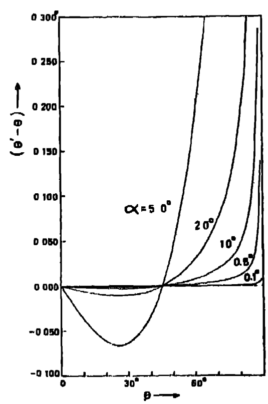


Figure 2. Variation of the change in $\theta$ with $\theta$ for various amounts of misalignment $\alpha$

$\theta$ below 45°, the effect of misalignment is to decrease the observed value of Bragg angles, the decrease being the maximum at $\theta = 26°$. For Bragg angles greater than 45° the effect is reversed, the difference $(\theta' - \theta)$ increasing rather sharply with $\theta$. Thus, unlike some other major systematic errors, error due to misalignment does not tend to zero as $\theta \rightarrow 90°$. This underlines the importance of good alignment of crystals, as it is the high-angle reflections on which much reliance is placed for an accurate determination of lattice parameters.

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